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Dentistry — Polymer-based composite machinable blanks

Médecine bucco-dentaire — Ébauches usinables en composite à base de polymères

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Foreword

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This document was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 9, *Dental CAD/CAM systems,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 55, *Dentistry,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Specific qualitative and quantitative test methods for demonstrating freedom from unacceptable biological hazards are not included in this document, but it is recommended that, for the assessment of possible biological hazards, reference should be made to ISO 10993-1 and ISO 7405.

Requirements for the materials properties of polymer-based composite machinable blanks are not included in this document, but these requirements will be included in a future edition of ISO 10477.

The test method to determine the bonding properties between blank and holding jig is not included in this document, but it is recommended to adopt the test procedure given in Annex C when measuring the bonding properties between blank and holding jig.

Dentistry — Polymer-based composite machinable blanks

1 Scope

This document specifies the characteristics of polymer-based composite machinable blanks with respect to the milling process and provides the test methods that address the clinical issues specific to those materials. In addition, this document specifies the items to be described on the packaging and materials, as well as descriptions to be included in the instructions for use.

The polymer-based composite machinable blanks covered in this document are blanks that are used for fabricating permanent dental restorative appliances (e.g. single crowns or inlays) by milling processes. They do not include large-sized blanks (e.g. discs) that allow for the fabrication of two or more units of crowns or bridges from one blank or materials for temporary use.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1942, *Dentistry — Vocabulary*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 4049:2019, *Dentistry — Polymer-based restorative materials*

ISO 6344-3, *Coated abrasives — Determination and designation of grain size distribution — Part 3: Microgrit sizes P240 to P5000*

ISO 6872:2015, *Dentistry — Ceramic materials*

ISO 8601-1, *Date and time — Representations for information interchange — Part 1: Basic rules*

ISO 18675:2022, *Dentistry — Machinable ceramic blanks*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
polymer-based composite machinable blank
piece of solid *polymer-based composite* (3.2) that is subjected to subtractive methods to remove material from the piece leaving the final desired part

3.2
polymer-based composite
polymer-based dental material including either organic filler or inorganic filler, or both fillers that have been treated by a coupling agent to ensure chemical bonding with the polymeric matrix

3.3
holding jig
mandrel
jig that attaches a composite resin block to be mounted for milling machine

4 Characteristics

4.1 Requirement

Measure five product blanks in accordance with the method described in 6.2. The dimensions of all blanks shall not be smaller than 0,25 mm nor larger than 1,00 mm than the size specified in Clause 8 d).

4.2 Recommendations

4.2.1 Machining damage

The machining damage of blanks should be evaluated in accordance with the methods described in 6.3.

4.2.2 Machinability

The machinability of blanks should be evaluated using the test method for merlon fracture test given in ISO 18675:2022, Clause 8.

4.2.3 Bonding properties between blank and holding jig

The bonding properties between blank and holding jig should be evaluated. An example of the test method for bonding properties is described in Annex C.

5 Sampling

The test sample shall consist of one or more packages for retail of one selected shade, corresponding to the purpose of the test, from a single batch and contain sufficient material to carry out the specified tests, plus an allowance for any necessary repetition of tests.

6 Test methods

6.1 General

Test specimens shall be prepared and tested at (23 ± 2) °C. The relative humidity shall be not less than 30 %.

6.2 Size of blanks

6.2.1 Apparatus

Micrometer, with an accuracy of 0,01 mm.

6.2.2 Procedure

Measure five blanks using a micrometer at the points where the size is specified by the manufacturer in the instructions for use in Clause 8 d).

6.3 Machining damage

6.3.1 General

Perform machining damage test in accordance with the method described in ISO 18675:2022, Clause 7. Specifically, measure the three-point flexural strength for the control specimen fabricated by cutting and grinding and for the machined specimen fabricated using a milling machine, and compare the results between these specimens.

6.3.2 Apparatus

6.3.2.1 Oven, set at (37 ± 1) °C.

6.3.2.2 Universal mechanical testing machine, capable of a crosshead speed of (1,0 ± 0,5) mm/min; for example, see ISO 7500-1.

6.3.2.3 Fixture for three-point bending, consisting of support rollers (1,5 mm to 2 mm in diameter, tolerances are ±0,2 mm) positioned with their centres (12,0 ± 0,1) mm apart. The load shall be applied at the midpoint between the supports by means of a third roller (1,5 mm to 2 mm in diameter, tolerances are ±0,2 mm). Rollers shall be made from hardened steel or other hard material having a hardness greater than 40 HRC (Rockwell C-scale) and have a smooth surface with a roughness less than 0,5 µm Ra. It is recommended to measure the actual spacing between the centres of the support rollers, l, to ensure it is (12,0 ± 0,1) mm.

6.3.2.4 Micrometer, with an accuracy of 0,01 mm.

6.3.2.5 CAD/CAM milling machine

6.3.3 Water

Water shall conform to ISO 3696:1987, Grade 2.

6.3.4 Preparation of test specimens

6.3.4.1 Test specimen dimensions

The width is: w = (4,0 ± 0,2) mm (dimension of the side at right angles to the direction of the applied load).

The thickness is: b = (1,2 ± 0,2) mm (dimension of the side parallel to the direction of the applied load).

Lengths shall be at least 2 mm longer than the test span (12,0 ± 0,1) mm.

When the edge chamfer is necessary, it shall be prepared in accordance with the method described in ISO 6872:2015, 7.3.1.2.1.

NOTE The dimensions of test specimen specified in this document are not verified for the measurement of flexural strength absolute values for composite resin materials. There are some published scientific papers in which flexural strength test of composite machinable blanks were performed at this size.

6.3.4.2 Test parameters

The test span is: l = (12,0 ± 0,5) mm (centre-to-centre distance between support rollers).

6.3.4.3 Control specimen

Prepare test specimens from blanks. Polish the surface of specimen until the required thickness is reached. Perform the final polishing using P1000 or finer silicon carbide waterproof abrasive paper. Confirm the absence of chipping in the centre portion.

Specimens shall be plane-parallel for bending test. Prepare five specimens. Store the specimen in water at (37 ± 1) °C for 7 d ± 4 h until the start of testing.

NOTE An example of detailed method is shown in Annex A.

6.3.4.4 Machined specimen

Fabricate test specimens of the abovementioned dimensions by the milling machine.

It is desirable to adopt a milling design and processing path that avoids bending test specimens during milling. It is also desirable to arrange appropriate supports in the milling design for this purpose. When setting the supports, ensure that the supports are located on the outer area of the test specimen for the three-point bending test.

After the machining process, cut off the support portion using an appropriate method (e.g. diamond disks).

Prepare five specimens having plane-parallel surfaces. Report the number of specimens that cannot be tested due to machining failure.

Store the specimen in water at (37 ± 1) °C for 7 d ± 4 h until the start of testing. The test specimens are subjected to the bending test without further surface polishing as is used for the control specimens.

NOTE Annex B gives an example of a specific milling design for test specimens from blanks.

6.3.5 Procedure

Measure the cross-sectional dimensions of each test specimen to ±0,01 mm. Then, place a test specimen centrally on the supports of the test machine so that the load is applied to a 4 mm wide face along a line perpendicular to the long axis of the test specimen and determine the maximum load required to break the test specimen. Use a crosshead speed of (1,0 ± 0,5) mm/min. Repeat the procedure with the remaining test specimens.

6.3.6 Expression of results

6.3.6.1 Calculation

Calculate the flexural strength, σ, in megapascals:

$$\sigma = \frac{3Fl}{2wb^2}$$

where

F is the maximum applied load, in newtons;

l is the distance, in millimetres, between the supports, i.e. 12 mm;

w is the width of the test specimen, in millimetres;

b is the thickness of the test specimen, in millimetres.

Calculate the mean and standard deviation of the flexural strength values for control and machined specimens, followed by the percentage of change in flexural strength of the machined group as compared with that of the control group.

6.3.6.2 Test report

Test report for machining damage should be in accordance with ISO 18675:2022, 7.3. The documentation of the test shall include at least the following information:

a) name of manufacturer, brand name, shade – if applicable;

b) size of the blank(s);

c) lot-number of the blank(s);

d) fabrication conditions of the control specimens including sectioning methodology and finishing of the surface;

e) length, width and height of the bar test specimens;

f) characterization (manufacturer, brand name, accuracy, etc.) of the micrometer gauge or another appropriate device used to perform all necessary dimension measurements;

g) characterization (manufacturer, brand name, accuracy, etc.) of the milling machine used to fabricate the specimens as a well as machining conditions (bur size, bur grit, feed rate if known) and software (manufacturer and version) used for machining;

h) instruments (manufacturer, brand-name, accuracy, etc.) used for mechanical testing and conditions of the test (such as crosshead speed, load cell);

i) flexural strength values of each specimen as well as mean and standard deviation of each group – control and machined;

j) percentage of change in flexural strength of the machined group as compared to the control group;

k) number of specimens that cannot be tested due to machining failure;

l) appropriate statistical analysis to determine significant differences;

m) International Standard used (including its year of publication);

n) any deviation from recommended test procedure and unusual features observed;

o) date of test.

7 Packaging and labelling

7.1 Packaging

The components of the polymer-based composite machinable blank shall be supplied in such containers that the contents are adequately protected and the quality of the polymer-based composite machinable blank is not adversely altered before the expiry date marked on the package and the container [see 7.2.2 f)].

7.2 Labelling

7.2.1 General

The relevant information shall be provided on the outer pack, polymer-based composite machinable blank or included in the instructions for use.

7.2.2 Labelling of outer pack

Each outer pack shall be labelled with at least the following information:

a) name and address of the manufacturer;

b) trade name of the polymer-based composite machinable blank;

c) shade(s) or description which can be related to the manufacturer's shade guide or that recommended by the manufacturer;

d) size dimensions and packaging quantity;

e) any special storage conditions;

f) expiry date when stored in accordance with the recommended conditions, expressed as year and month according to ISO 8601-1;

g) lot number (batch code);

h) the word “radio-opaque” if the material is claimed to be in accordance with the requirements of ISO 4049:2019, 5.5.

Additional information can be included at the discretion of the manufacturer.

7.2.3 Labelling of polymer-based composite machinable blank

Machinable blank (the resin part or holding jig) shall be labelled with at least the following information:

a) statements allowing identification of the product name or product;

b) shade(s) or description which can be related to the manufacturer's shade guide or that recommended by the manufacturer;

c) lot number (batch code).

8 Instructions for use

Instructions for use, together with the description of the product, shall be provided by the manufacturer for the user and accompany each set or package of polymer-based composite machinable blank with at least the following information:

a) name and address of either the manufacturer or the distributor, or both;

b) trade name of the polymer-based composite machinable blank;

c) range of application;

d) dimensions of blanks;

e) range of dimensions of inorganic filler particles and the percentage by volume of total inorganic filler;

f) processing (CAD/CAM milling) conditions (recommended milling machines, etc., as needed);

g) information about adhesive (either treatment method and recommended adhesives or cements, or both); if adhesion strength is to be specified in the instruction for use, indicate specified adhesives and test methods;

h) recommended procedure for finishing and polishing, and the polishing agent to be used;

i) any special storage conditions;

j) if radio-opacity is claimed, this shall be determined in accordance with ISO 4049:2019, 7.14 and the value stated in the instructions; if certain shades amongst others that are radio-opaque are not radio-opaque, this shall be stated; if the radio-opacity value of another shade or shades is more than twice the "universal" shade value, this shall be stated, and the manufacturer shall give an indication, either quantitatively or qualitatively, of the differences; an explanation of the radio-opacity value shall be included, for example, aluminium has a radio-opacity equivalent to that of dentine; thus 1 mm of material having a radio-opacity equivalent to 1 mm of aluminium has a radio-opacity equivalent to that of dentine and 2 mm of aluminium is equivalent to enamel;

k) date of issue of instructions for use.

Additional information can be included at the discretion of the manufacturer.

Annex A
(informative)

Preparation method of control specimen for flexural strength

A.1 General

This annex presents the method for preparing flexural strength test specimens without edged chamfer by cutting of them from blanks. The bending test specimens prepared under this method can be used as control specimens for machining damage tests.

This annex illustrates the method for making eight test specimens with dimensions of 4 mm × 1,2 mm × 14 mm from a blank measuring 14,5 mm × 14,5 mm × 18 mm.

A.2 Apparatus

A.2.1 Cutting machine.

The cutting machine should be equipped with a holding unit capable of fixing the block and a water-irrigation function and to allow preparation of test specimens with parallel planes using a diamond blade.

EXAMPLE The following model is an example of a cutting machine suitable for the purpose[1)].

Model name: IsoMet® 1000 (BUEHLER)

Cutter part No.: IsoMet® Diamond Wafering Blades

Grit grade: 15LC Diamond, 15HC Diamond

Arbor Size: 12,7 mm, diameter 102 mm × blade thickness 0,3 mm, model No.: 11–4254

Arbor Size: 12,7 mm, diameter 152 mm × blade thickness 0,5 mm, model No.: 11–4276

A.2.2 Micrometer, with an accuracy of 0,01 mm.

A.2.3 Silicon carbide abrasive paper, waterproof and grit sizes of silicon carbide of P1000 and P2000 in accordance with ISO 6344-3.

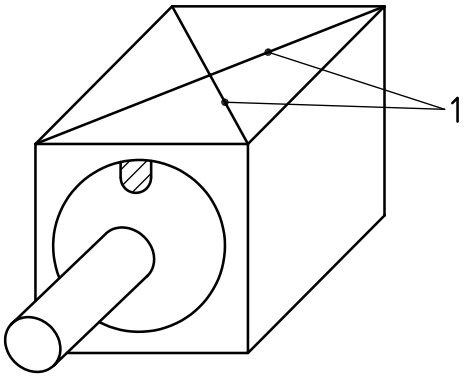
A.3 Procedure

A.3.1 Cutting of test specimens in the longitudinal (length) direction

Choose any of the 14,5 mm × 18 mm sides of the blank, and draw lines using a permanent oil-based marker pen or the like to prepare the blank for cutting the 4 mm wide plane of the test specimen (see Figure A.1).

Mounting the holding jig of the blank to the cutting machine's holding unit, cut the blank along the marked lines to obtain a length of at least 14,2 mm (see Figure A.2).

1) IsoMet® is a product name of BUEHLER, 41 Waukegan Road Lake Bluff, IL, 60044-1699 USA. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement of this system by ISO.



Key

1 drawing lines

Figure A.1 — Specification for the position of the 4 mm width plane of the test specimen

Dimensions in millimetres

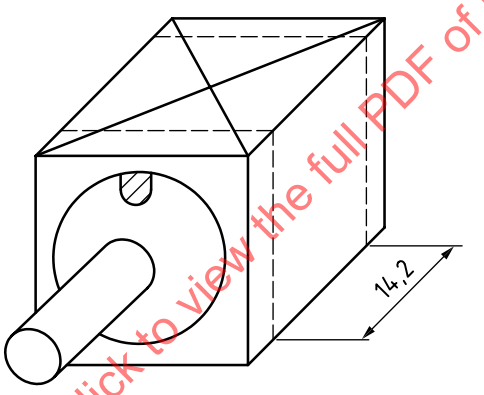


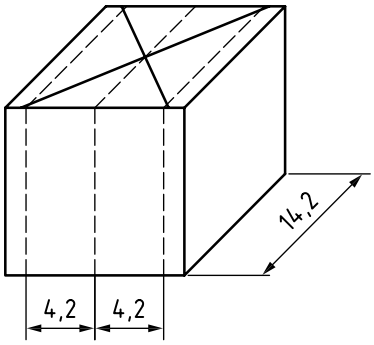
Figure A.2 — Cutting of test specimens in the longitudinal (length) direction

A.3.2 Cutting of test specimens in the width, *w*, direction

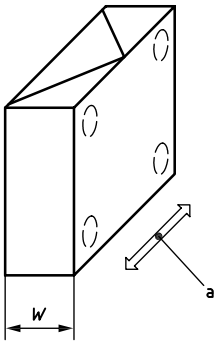
Mount the long side of the blank to the cutting machine's holding unit and cut the long axis direction under running water to 4,2 mm thickness (see Figure A.3). Next, using P1000 and then P2000 waterproof abrasive paper, polish the surface described in Figure A.3 and its opposite surface in the long-axis direction, so that the width of the specimen, *w,* measured at four sites is within the range of (4,0 ± 0,2) mm, and differences among the measured values are within 0,05 mm.

After polishing with abrasive paper of at least P1000, visually check for any flaws caused during cutting or polishing.

Dimensions in millimetres



a) Cutting planes



b) Polishing direction and specimen width measurement points

[a] The unfilled arrow in the figure shows the polishing direction.

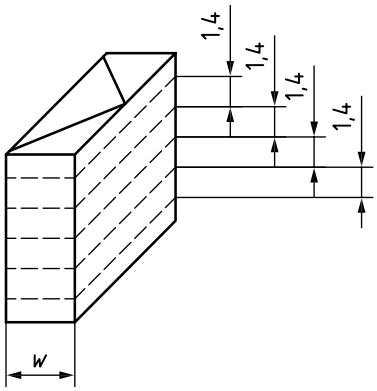
NOTE The dotted circles in Figure A.3 b) indicate the specimen width measurement points.

Figure A.3 — Cutting of test specimens in the width, *w*, direction

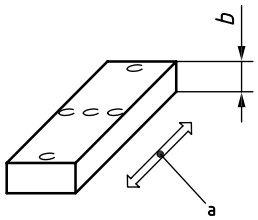
A.3.3 Cutting of test specimens in the thickness, *b*, direction

Fix the longitudinal (length) side onto the cutting machine's holding unit and cut it under running water to obtain a thickness of about 1,4 mm (where indicated by dotted lines) (see Figure A.4). Subsequently, polish the cut pieces using a waterproof abrasive paper of at least P1000 or finer, and perform dry polishing in the long-axis direction on the length-width plane (both the front and back sides) and the length-thickness plane (both the front and back sides). In the short-axis direction, the thickness measured at three points, including the centre, shall be within the range of (1,2 ± 0,2) mm, and differences between the measured values should be within 0,01 mm. In the long-axis direction, the thickness measured at three points including the centre shall be within the range of (1,2 ± 0,2) mm, and differences between the measured values should be within 0,02 mm (see Figure A.4).

Dimensions in millimetres



a) Cutting planes



b) Polishing direction and thickness measurement points

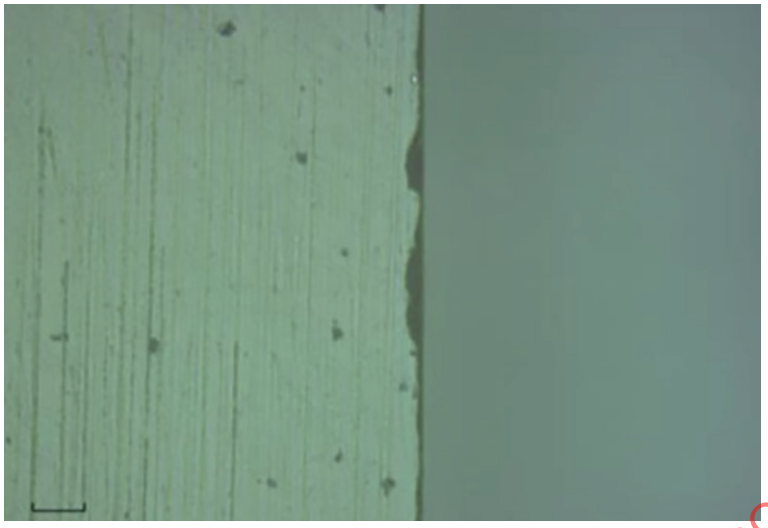
[a] The unfilled arrow in the figure shows the polishing direction.

NOTE The dotted circles in Figure A.4 b) indicate the thickness measurement points.

Figure A.4 — Cutting of test specimens in the thickness, *b*, direction

After polishing using abrasive paper of P1000 or finer, eliminate specimens with any flaws visually detected.

Verify that there are no evident chips on the edge of the test specimen (see Figure A.5).



a) Test specimen with chipped edges

b) Test specimen without chipped edges

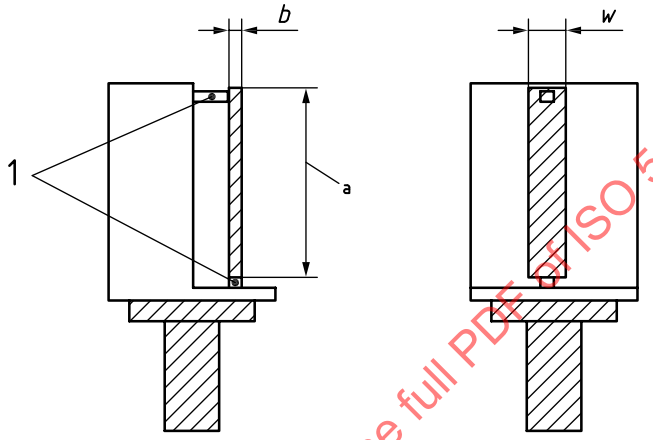
NOTE The length of indicated line means the scale of 100 µm.

Figure A.5 — Edge of the test specimen

Annex B
(informative)

Milling design of test specimen for machining damage

Figure B.1 presents an example of milling design for preparing bending-test specimens used to determine machining damage in blanks.



Key

w width, $w = (4 \pm 0{,}2)$ mm

b thickness, $b = (1{,}2 \pm 0{,}2)$ mm

1 support

[a] The arrow in the figure shows the length of the test specimen.

Figure B.1 — Example of milling design

Annex C
(informative)

Test method to determine the bonding properties between blank and holding jig

C.1 General

The blank should not peel off or fall out of the holding jig during the milling process. In this annex, debonding test and impact test are presented as methods for measuring the bonding properties between blank and holding jig.

C.2 Debonding test

C.2.1 General

This clause describes the method for measuring the bonding property between blank and holding jig by performing a debonding test. For measurement, the holding jig is set onto the debonding test fixture so as to apply a load perpendicular to the longitudinal axis to the holding jig (see Figure C.1). This test is a type of peel test and simulating the initial step of touching the milling bar on the blank.

C.2.2 Apparatus

C.2.2.1 Universal mechanical testing machine, capable of a crosshead speed of (1,0 ± 0,5) mm/min; for example, see ISO 7500-1.

C.2.2.2 Load applying blade, made of hard material (e.g. metal), of which the portion contacting the blank is shaped as a rod with a diameter of (2,0 ± 0,2) mm and a length equal to or greater than the blank width. The blade should be movable in the direction parallel to the bonding surface between the holding jig and the blank by pressing from above the blank.

C.2.2.3 Debonding test fixture, which is a jig fixing device made of hard material (e.g. metal and plastic) capable of setting the bonding surface between the holding jig and blank parallel to the direction of the blade movement.

C.2.3 Procedure

Set the holding jig onto the debonding test fixture and mount it so that the blade can apply a load perpendicular to the longitude axis of the holding jig.

At this step, position the blade at the point of the total length of the block minus (2,0 ± 0,5) mm from the bonding surface between blank and holding jig.

Apply a load to the test specimen at a crosshead speed of (1,0 ± 0,5) mm/min (debonding test). Record the load at the debonding.